PREFACE

In July 1976, the Preliminary Interim Method for Deter-
mining Asbestos in Water was issued by the U.S. Environmental
Protection Agency's Environmental Research Laboratory in
Athens, Georgia. That method was perceived as representing
the current state-of-the-art in asbestos analytical method-
ology. The objective of writing the method was to present a
procedure that analytical laboratories could follow that would
result in a better agreement of analytical results. Since
that time, a significant amount of additional experimental
work has generated data that provide the basis for a more
definitive method than was possible previously.

This revised Interim Method reflects the improvements
that have been made in asbestos analytical methodology since
the initial procedure was drafted. The general approach to
the analytical determination, however, remains the same as
previously outlined. That is, asbestos fibers are separated
from water by filtration on a sub-micron pore size membrane
filter. The asbestos fibers are then counted, after
dissolving the filter material, by direct observation in a
transmission electron microscope.

The major change in the initial procedure is the elimina-
tion of the condensation washer as a means of sample prepara-
tion. Intra- and inter-laboratory precision data for the
method are presented. Also, a suggested statistical evalua-
tion of grid fiber counts is included.

FOREWORD

Nearly every phase of environmental protection depends on a capability to identify and measure specific pollutants in the environment. As part of this Laboratory's research on the occurrence, movement, transformation, impact, and control of environmental contaminants, the Analytical Chemistry Branch develops and assesses new techniques for identifying chemical constituents of water and soil.

The widespread use of asbestos-containing materials gives rise to concern about exposure of the general population to low level concentrations in air, water supplies, and food. Although hazards associated with the inhalation of asbestos at high concentrations are recognized, the health significance of ingested particles is not fully understood. An integral part of the U.S. Environmental Protection Agency's research into the health effects of exposure to this naturally occurring mineral is the development of an accurate analysis method for determining its presence in water. To this end, this report presents an interim EPA procedure for analytical laboratories to follow that would result in better agreement of analytical results.

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ABSTRACT

An interim electron microscope (EM) procedure for measuring the concentration of asbestos in water samples is described. The main features of the method include filtering the samples through a sub-micron polycarbonate membrane filter, examining an EM specimen grid in a transmission electron microscope (TEM), and verifying fiber identity by selected area electron diffraction.

This interim method is a revision of the procedure issued in 1976 and reflects the improvements that have been made in asbestos analytical methodology since that time.

This report covers a period from July 1976 to December 1978 and work was completed as of December 1978.

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FIGURES

TABLES

INTERIM METHOD
FOR DETERMINING ASBESTOS IN WATER

1.   Scope and Application

1.1   This method is applicable to drinking water and
      water supplies.

1.2   The method determines the number of asbestos fibers
      per liter, the size (length and width) of the
      fibers, the size distribution, and the total mass.
      The method distinguishes chrysotile from amphibole
      asbestos. The detection limits are variable and
      depend upon the amount of total extraneous particu-
      late matter in the sample as well as the contamina-
      tion level in the laboratory environment. Under
      favorable circumstances, 0.01 million fibers per
      liter (MFL) can be detected. The detection limit
      for total mass of asbestos fibers is also variable
      and depends upon the fiber size and size distribu-
      tion in addition to the factors affecting the total
      fiber count. The detection limit under favorable
      conditions is in the order of 0.1 nanogram per
      liter (ng/L).

1.3   The method is not intended to furnish a complete
      characterization of all the fibers in water.

1.4   It is beyond the scope of this method to furnish
      detailed instruction in electron microscopy,
      electron diffraction, or crystallography. It is
      assumed that those using this method will be suffi-
      ciently knowledgeable in these fields to understand
      the methodology involved.

2.   Summary of Method

2.1   A variable, known volume of water sample is
      filtered through a 0.1 micrometer (μm) Nuclepore
      filter to trap asbestos fibers and the filter is
      then carbon coated. A small portion of the carbon
      coated filter with deposited fibers is placed on an
      electron microscope grid and the filter material is
      removed by gentle solution in organic solvent. The

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material remaining on the electron microscope grid
is examined in a transmission microscope at a
magnification of about 20,000X. The asbestos
fibers are identified by their morphology and
electron diffraction patterns and their lengths and
widths are measured. The total area examined in
the electron microscope is determined and the
number of asbestos fibers in this area is counted.
The concentration in MFL is calculated from the
number of fibers counted, the amount of water
filtered, and the ratio of the total filtered area/
sampled filter area. The mass per liter is calcu-
lated from the assumed density and the volume of
the fibers.

3.   Definitions

Asbestos - A generic term applied to a variety of
        commercially useful fibrous silicate minerals of
        the serpentine or amphibole mineral groups.

Fiber - Any particulate that has parallel sides and a
        length/width ratio greater than or equal to 3:1.

Aspect Ratio - The ratio of length to width.

Chrysotile - A nearly pure hydrated magnesium silicate,
        the fibrous form of the mineral serpentine,
        possessing a unique layered structure in which the
        layers are wrapped in a helical cylindrical manner
        about the fiber axis.

Amphibole Asbestos - A double chain fibrous silicate
        mineral consisting of Si_4O_{11}, laterally linked by
        various cations such as aluminum, calcium, iron,
        magnesium, and sodium. Amphibole asbestos consists
        of crocidolite and amosite (the fibrous form of
        cummingtonite-gruenerite), and the fibrous forms of
        tremolite, actinolite, and anthophyllite. These
        minerals consist of or contain fibers formed
        through natural growth processes. Mineral frag-
        ments that conform to the definition of a fiber and
        that are formed through a crushing and milling
        process are analytically indistinguishable from the
        naturally formed fibers by this method.

Detection Limit - The calculated concentration in MFL,
        equivalent to one fiber above the background or
        blank count (Section 8.6).

Statistically Significant - Any concentration based upon a total fiber count of 5 or more in 20 grid squares.

4.   Sample Handling and Preservation

It is beyond the scope of this procedure to furnish detailed instructions for field sampling; the general principles of obtaining water samples apply. Some specific considerations apply to asbestos fibers, however, because they are a special type of particulate matter. These fibers are small, and in water range in length from 0.1 μm to 20 μm or more. Because of the range of size there may be a vertical distribution of particle sizes. This distribution will vary with depth depending upon the vertical distribution of temperature as well as local meteorological conditions. Sampling should take place according to the objective of the analysis. If a representative sample of a water supply is required, a carefully designated set of samples should be taken representing the vertical as well as the horizontal distribution and these samples should be composited for analysis.

4.1   Containment Vessel

The sampling container shall be a clean, screw-capped, polyethylene bottle capable of holding at least 1 liter. The bottle should be rinsed at least two times with the water that is being sampled prior to sampling.

4.2   Quantity of Sample

A minimum of approximately 1 liter of water is required. Leave air space at the top of the container to allow for shaking the sample. It is desirable to obtain two samples from one location.

4.3   Sample Preservation

No preservatives should be added during sampling and the addition of acids should be particularly avoided. If the sample cannot be filtered in the laboratory within 48 hours of its arrival, sufficient amounts (1 milliliter per liter of sample) of a 2.71% solution of mercuric chloride to give a final concentration of 20 ppm of Hg may be added to prevent bacterial growth.

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NOTE 1: It has been reported that the growth of algae in water samples can be prevented by storing the samples in the dark.

NOTE 2: Refrigeration of samples at about 5°C minimize bacterial and algal growth.

5. Interferences

5.1 Misidentification

The guidelines set forth in this method for counting fibrous asbestos require a positive identification by both morphology and crystal structure as shown by an electron diffraction pattern. Chrysotile asbestos has a unique tubular structure, usually showing the presence of a central canal, and exhibits a unique characteristic electron diffraction pattern. Although halloysite fibers may show a streaking similar to chrysotile, they do not exhibit chrysotile's characteristic triple set of double spots or 5.3A layer line. It is highly improbable that a non-asbestiform fiber would exhibit the distinguishing chrysotile features. Although amphibole fibers exhibit characteristic morphology and electron diffraction patterns, they do not have the unique properties exhibited by chrysotile. It is possible, there-fore, though not probable for misidentification to take place.

It is important to recognize that a significant variable fraction of both chrysotile and amphibole asbestos fibers do not exhibit the required confirmatory electron diffraction pattern. This absence of diffraction is attributable to unfavor-able fiber orientation and fiber sizes. The results reported will be low, therefore, as compared to the absolute number of asbestos fibers that are present.

5.2 Obscuration

If large amounts of other materials are present, some small asbestos fibers may not be observed because of physical overlapping. This will result in low values for the reported asbestos content.

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5.3 Contamination

Although contamination is not strictly considered to be an interference, it is an important source of erroneous results, particularly for chrysotile. The possibility of contamination, therefore, should always be a consideration.

5.4 Freezing

The effect of freezing on asbestos fibers is not known but there is reason to suspect that fiber breakdown could occur and result in a higher fiber count than was present in the original sample. Therefore, the sample should be transported to the laboratory under conditions that would avoid freezing.

6. Equipment and Apparatus

6.1 Specimen Preparation Laboratory

The ubiquitous nature of asbestos, especially chrysotile, demands that all sample preparation steps be carried out to prevent the contamination of the sample by airborne or other source of asbestos. The prime requirement of the sample preparation laboratory is that it be sufficiently free from asbestos contamination that a specimen blank determination using 200 ml of asbestos-free water yields no more than 2 fibers in 20 grid squares of a conventional 200 mesh electron microscope grid.

In order to achieve this low level of contamination, the sample preparation area should be a separate conventional clean room facility. The room should be operated under positive pressure and have incorporated electrostatic precipitators in the air supply to the room, or as an alternative, absolute (HEPA) filters. No asbestos floor or ceiling tiles, transite heat-resistant boards, or asbestos insulation should be used in construction. Work surfaces should be stainless steel or Formica or equivalent. A laminar flow hood should be provided for sample manipulation. Disposable plastic laboratory coats and disposable overshoes are recommended. Alternatively, new shoes for all operators should be provided and retained for clean room use only. A mat (Tacky Mat, Liberty Industries, 589 Deming Road, Berlin, Connecticut 06037, or equivalent) should be placed at the

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entrance to the room to trap any gross contamination inadvertently brought into the room on contaminated shoes. Normal electrical and water services, including a distilled water supply should be provided. In addition, a source of ultra-pure water from a still or filtration-ion exchange system is desirable.

6.2   Instrumentation

6.2.1   Transmission Electron Microscope. A transmission electron microscope that operates at a minimum of 80 kV and has a resolution of better than 1.0 nm and a magnification range of 300 to 100,000 is required. If the upper limit is not attainable directly it may be attained through the use of auxiliary optical viewing. It is mandatory that the instrument be capable of carrying out selected area electron diffraction (SAED) on an area of about 0.3 μm^2. The viewing screen shall have either a millimeter scale, concentric circles of known radii, or other devices to measure the length and width of the fiber. Most modern transmission microscopes meet the requirements for magnification and resolution.

An energy-dispersive X-ray spectrometer is useful for the identification of suspected asbestiform minerals; this accessory to the microscope, however, is not mandatory.

6.2.2   Data Processor. The large number of repetitive calculations make it convenient to use computer facilities together with relatively simple computer programs.

6.2.3   Vacuum Evaporator. A vacuum evaporator is required for depositing a layer of carbon on the Nuclepore filter and for preparing carbon coated grids.

6.2.4   Low Temperature Plasma Asher. An asher is used for the removal of organic material (including the filter) from samples containing so much organic matter that asbestos fibers are obscured. The sample chamber should be at least 10 cm in diameter.

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6.3 Apparatus, Supplies and Reagents

6.3.1 Jaffe Wick Washer. The Jaffe Wick Washer for dissolving Nuclepore filter is described in 8.3.1, and is illustrated in Figure 1.

6.3.2 Filtering Apparatus. A 47-mm funnel (Cat No. XX1504700, Millipore Corporation, Order Service Dept., Bedford, MA 01730) or equivalent is used to filter water samples. A 25-mm funnel (Millipore Cat No. XX1002500) or equivalent is used to filter dispersed ash samples.

6.3.3 Vacuum Pump. A pump, for use in sample filtration, should provide vacuum up to about 500 mm of mercury.

6.3.4 EM Grids. Grids of 200-mesh copper or nickel covered with formvar film for use with the Nuclepore-Jaffe sample preparation method are required. These grids may be purchased from manufacturers of electron microscopic supplies or prepared by standard electron microscopic grid preparation procedures. Finder grids may be substituted and are useful if the re-examination of a specific grid opening is desired.

6.3.5 Membrane Filters.

47-mm diameter Millipore membrane filter, type HA, 0.45-μm pore size. Used as a Nuclepore filter support on top of glass frit.

47-mm diameter Nuclepore membrane filter; 0.1-μm pore size (Nuclepore Corp., 7035 Commerce Circle, Pleasanton, CA 94566). Used to filter the water sample.

25-mm diameter Millipore membrane filter, type HA; 0.45-μm pore size. Used as Nuclepore filter support on top of glass frit.

25-mm diameter Nuclepore membrane filter; 0.1-μm pore size. Used to filter dispersed ashed Nuclepore filter.

Screen Support
with Grid

Petri Dish

Ridge

Glass Slides

Layer of
Filter Papers

A

Nuclepore Filter

Carbon

Carbon
Chloroform

Formvar

Grid

Grid

B
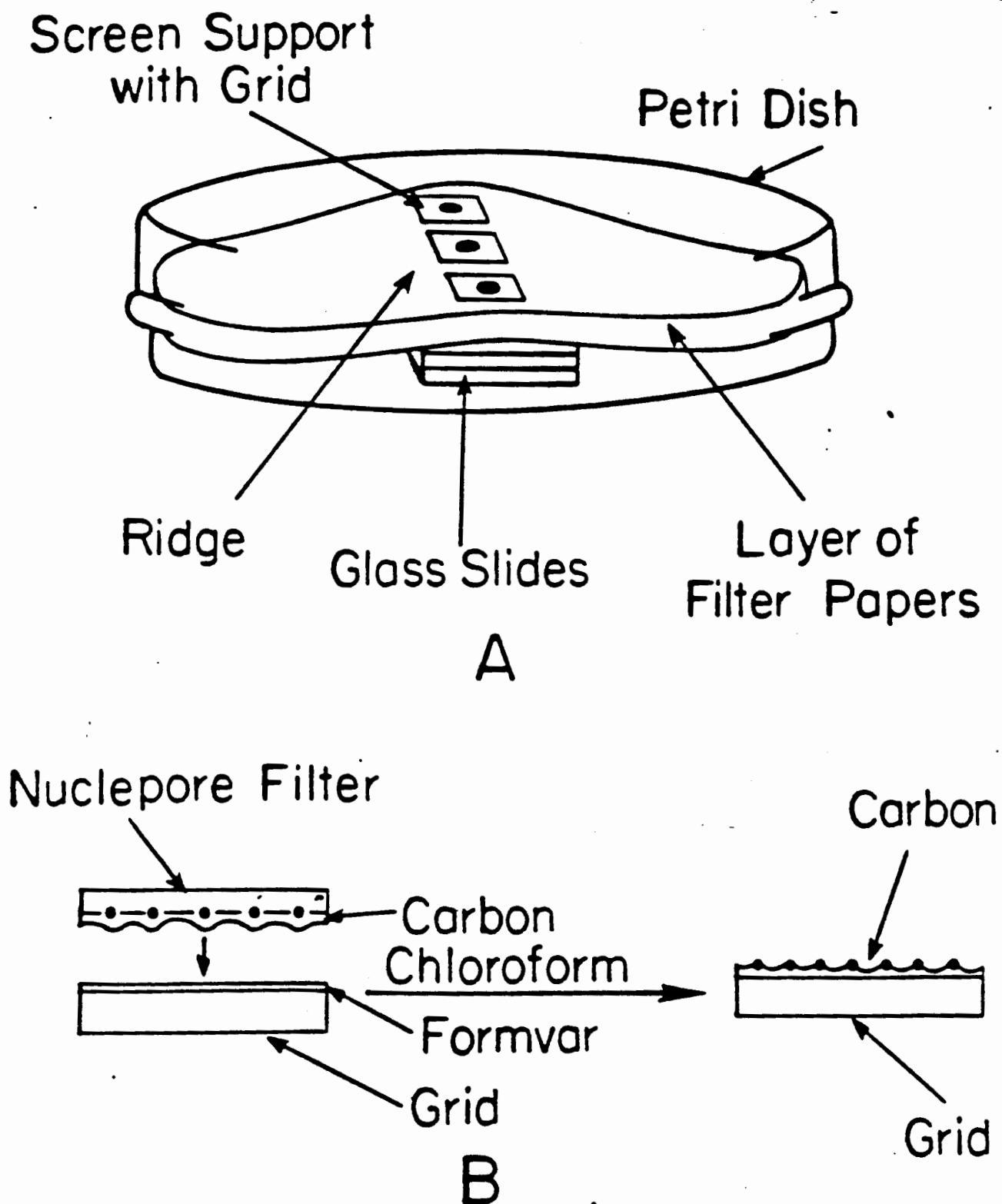
Figure 1. Modified Jaffe Wick Method.
          A. Washing Apparatus
          B. Washing Process

6.3.6   Glass Vials. 30-mm diameter x 80-mm long.
        Used to hold filter during ashing.

6.3.7   Glass Slides. Used to support Nuclepore
        filter during carbon evaporation.

6.3.8   Scalpels.

6.3.9   Scissors.

6.3.10 Tweezers. Several pairs are needed for the
        many handling operations.

6.3.11 Double-sided Tape. Used to hold filter
        section flat on glass slide while carbon
        coating is applied.

6.3.12 Disposable Petri Dishes, 50-mm diameter.
        Used for storing membrane filters.

6.3.13 Static Eliminator, 500 microcuries Po-210.
        (Nuclepore Cat. No. V090POL00101) or equiv-
        alent. Used to eliminate static charges
        from membrane filters.

6.3.14 Carbon Rods. Spectrochemically pure, 1/8
        in. dia., 3.6 mm x 1.0 mm neck. Used for
        carbon coating.

6.3.15 Carbon rod sharpener. (Cat. No. 1204,
        Ernest F. Fullam, Inc., P. O. Box 444,
        Schenectady, NY 12301) or equivalent. Used
        for sharpening carbon rods to a neck of
        specified length and diameter.

6.3.16 Ultrasonic Bath. (50 watts, 55 kHz). Used
        for dispersing ashed sample and for general
        cleaning.

6.3.17 Graduated Cylinder, 500 ml.

6.3.18 Spot Plate.

6.3.19 10-µl Microsyringe. Used for administering
        drop of solvent to filter section during
        sample preparation.

6.3.20 Carbon Grating Replica, 2160 lines/mm.
        Used for calibration of EM magnification.

6.3.21 Filter Paper. S & S #589 Black Ribbon (9-cm circles) or equivalent absorbent filter paper. Used for preparing Jaffe Wick Washer.

6.3.22 Screen supports (copper or stainless steel) 12 mm x 12 mm, 200 mesh or equivalent. Used to support specimen grid in Jaffe Wick Washer.

6.3.23 Chloroform. Spectro grade, doubly distilled. Used for dissolving Nuclepore filters.

6.3.24 Asbestos. Chrysotile (Canadian), Crocidolite, Amosite. UICC (Union Internationale Contre le Cancer) Standards. Available from Duke Standards Company, 445 Sherman Avenue, Palo Alto, CA 94306.

6.3.25 Petri Dish. Glass dish (100 mm diameter x 15 mm high). Used for modified Jaffe Wick Washer.

6.3.26 Alconox. (Alconox, Inc., New York, NY 10003) or equivalent. Used for cleaning glassware. Add 7.5 g Alconox to a liter of distilled water.

6.3.27 Parafilm. (American Can Company, Neenah, WI) or equivalent. Used as protective covering for clean glassware.

6.3.28 Pipets. Disposable, 5 ml and 50 ml pipets are required.

6.3.29 Distilled or Deionized Water. Filter if necessary through 0.1-µm Nuclepore filter for making up all reagents, for final rinsing of glassware, and for preparing blanks.

6.3.30 Mercuric chloride, 2.71% solution w/v. Used as sample preservative. See 4.3. Add 5.42 g of reagent grade mercuric chloride ($HgCl_2$) to 100 ml distilled water and dissolve by shaking. Dilute to 200 ml with additional water. Filter through 0.1-µm Nuclepore filter paper before using.

7. Preparation of Standards

Reference standard samples of asbestos that can be used
for quality control for a quantitative analytical method
are not available. It is, however, necessary for each
laboratory to prepare at least two suspensions, one of
chrysotile and another of a representative amphibole.
These suspensions can then be used for intra-laboratory
control and to furnish standard morphology photographs
and diffraction patterns.

7.1 Chrysotile Stock Suspension.

Grind about 0.1 g of UICC chrysotile to a powder in
an agate mortar. Transfer 10 mg to a clean 1-liter
volumetric flask, add several hundred ml of
filtered distilled water containing 1 ml of a stock
mercuric chloride solution and then make up to 1·
liter with filtered distilled water. To prepare a
working solution, transfer 10 ml of the above
suspension to another 1-liter flask, add 1 ml of a
stock mercuric chloride solution and make up to 1
liter with filtered distilled water. This suspen-
sion contains 100 µg per liter. Finally transfer 1
ml of this suspension to a 1-liter flask, add 1 ml
of a stock mercuric chloride solution and make up
to volume with filtered distilled water. The final
suspension will contain 5 to 10 MFL and is suitable
for laboratory testing.

7.2 Amphibole Stock Suspension.

Prepare amphibole suspensions from UICC amphibole
samples as in Section 7.1.

7.3 Identification Standards.

Prepare electron microscopic grids containing the
UICC asbestos fibers according to Section 8 and
obtain representative photographs of each fiber
type and its diffraction pattern for future
reference.

8. Procedure

8.1 Filtration.

The separation of insoluble material, including
asbestiform minerals, through filtration and subse-
quent deposition on a membrane filter is a critical
step in the procedure. The objective of the

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filtration is not only to separate, but also to distribute the particulate matter uniformly such that discrete particles are deposited with a minimum of overlap.

The volume filtered will range from 50 to 500 ml. In an unknown sample, the volume can not be specified in advance because of the presence of variable amounts of particulate matter. In general, sufficient sample is filtered such that a very faint stain can be observed on the filter medium. The maximum loading that can be tolerated is 20 $\mu g/cm^2$, or about 200 μg on a 47-mm diameter filter; 5 $\mu g/cm^2$ is near optimum. If the total solids content is known, an estimate of the maximum volume tolerable can be obtained. In a sample of high solids content, where less than 50 ml is required, the sample should be diluted with filtered distilled water so that a minimum total of 50 ml of water is filtered. This step is necessary to allow the insoluble material to deposit uniformly on the filter. The filtration funnel assembly must be scrupulously cleaned before each filtration. The filtration should be carried out in a laminar flow hood.

NOTE: The following cleaning procedure has been found to be satisfactory.

Wash each piece of glassware three times with distilled water. Following manufacturer's recommendations, use the ultrasonic bath with an Alconox-water solution to clean all glassware. After the ultrasonic cleaning, rinse each piece of glassware three times with distilled water. Then rinse each piece three times with deionized water that has been filtered through 0.1-μm Nuclepore filter. Dry in an asbestos-free oven. After the glassware is dry, seal openings with parafilm.

> a. Assemble the vacuum filtration apparatus incorporating the 0.1-μm Nuclepore backed with 0.45-μm Millipore filter. See 8.3.2.

> b. Vigorously agitate the water sample in its container. Treatment of the sample in an ultrasonic bath may be required to evenly disperse the particulate material.

c. If the required filtration volume can be estimated, either from turbidity estimates of suspended solids or previous experience, immediately withdraw the proper volume from the container and add the entire volume to the 47-mm diameter funnel. Apply vacuum sufficient for filtration but gentle enough to avoid the formation of a vortex. If a completely unknown sample is being analyzed, a slightly modified procedure must be followed. Pour 500 ml of a well-mixed sample into a 500-ml graduated cylinder and immediately transfer the entire contents to the prepared vacuum filtration apparatus. Apply vacuum gently and continue suction until all of the water has passed through the filter. If the resulting filter appears obviously coated or discolored, ' another filter should be prepared in the same manner, but this time using only 200 or 100 ml of sample.

NOTE 1: Do not add more water after filtration has started and do not rinse the sides of the funnel.

NOTE 2: Nuclepore filter is basically a hydrophobic material. The manufacturer applies a detergent to the surface of the filter in order to render it hydrophilic; this process, however, does not appear to be entirely satisfactory in some batches. Pretreatment of the filter in a low temperature asher at 10 watts for 10 seconds can be used to render the surface of the filter hydrophilic. This process will significantly decrease the islands of sparse deposit that are frequently observed.

d. Disassemble the funnel, remove the filter, and dry it in a covered petri dish.

8.2   Preparation of Electron Microscope Grids.

Preparation of the grid for examination in the microscope is a critical step in the analytical procedure. The objective is to remove the organic filter material from the asbestos fibers with minimum loss and movement and with minimum breakage of the grid support film.   •

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If the sample contains large amounts of organic
matter that interfere with fiber counting and
identification a preliminary ashing step is
required. See 8.5.

8.3   Nuclepore Filter, Modified Jaffe Wick Technique.

8.3.1   Preparation of Modified Jaffe Washer

Place three glass microscope slides (75 mm
x 22 mm) one on top of the other in a petri
dish (100 mm x 15 mm) along a diameter.
Place 14 S & S #589 Black Ribbon filter
papers (9-cm circles) in the petri dish
over the stack of microscope slides. Place
three copper mesh screen supports (12 mm x
12 mm) along the ridge formed by the stack
of slides underneath the layer of filter.
papers. Place an EM specimen grid on each
of the screen supports. See Fig. 1.

NOTE: A stack of 30 to 40 S & S filters
(7-cm circle), or equivalent, can be
substituted for the 14 filters and micro-
scope slides in preparing the Jaffe washer.

8.3.2   Vacuum Filtration Unit

Assemble the vacuum filtration unit. Place
a 0.45-μm Millipore filter type HA on the
glass frit and then position a 0.1-μm
Nuclepore filter, shiny side up, on top of
the Millipore filter. Apply suction to
center the filters flat on the frit.
Attach the filter funnel and shut off the
suction.

8.3.3 - Sample Filtration

See 8.1.

8.3.4   Sample Drying

Remove the filter funnel and place the
Nuclepore filter in a loosely covered petri
dish to dry. The petri dish containing the
filter may be placed in an asbestos-free
oven at 45° C for 30 minutes to shorten
the drying time.

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Alternatively, the Nuclepore filter section
may be mounted on a glass slide prior to
drying the filter.

8.3.5   Selection of Section for Carbon Coating

Using a small pair of scissors or sharp
scalpel, cut out a rectangular section of
the Nuclepore filter. The minimum approxi-
mate dimensions should be 15 mm long and 3
mm wide. Avoid selection near the perim-
eter of the filtration area.

8.3.6   Carbon Coating the Filter

Tape the two ends of the selected filter
section to a glass slide using double-sided
tape. Take care not to stretch the filter
section. Identify the filter section using
a china marker on the slide. Place the
glass slide with the filter section into
the vacuum evaporator. Insert the necked
carbon rod and, following manufacturer's
instructions, obtain high vacuum. Evapo-
rate the neck, with the filter section
rotating, at a distance of approximately
7.5 cm from the filter section to obtain a
30 to 50 nm layer of carbon on the filter
paper. Evaporate the carbon in several
short bursts rather than continuously to
prevent overheating the surface of the
Nuclepore filter.

NOTE 1: Overheating the surface tends to
crosslink the plastic, rendering the filter
dissolution in chloroform difficult.

NOTE 2: The thickness of the carbon film
can be monitored by placing a drop of oil
on a porcelain chip that is placed at the
same distance from the carbon electrodes as
the specimen. Carbon is not visible in the
region of the oil drop thereby enabling the
visual estimate of the deposit thickness by
the contrast differential.

8.3.7   Grid Transfer

Remove the filter from the vacuum evapo-
rator and cut out three sections somewhat
less than 3 mm x 3 mm and such that the

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square of Nuclepore fits within the circumference of the grid. Pass each of the filter sections over a static eliminator and then place each of the three sections carbon-side down on separate specimen grids previously placed in the modified Jaffe Washer. Using a microsyringe, place a 10-μl drop of chloroform on each filter section resting on a grid and then saturate the filter pad until pooling of the solvent occurs below the ridge formed by the glass slides inserted under the layer of filter papers. Place the cover on the petri dish and allow the grids to remain in the washer for approximately 24 hours. Do not allow the chloroform to completely evaporate before the grids are removed. To remove the grids from the washer, lift the screen support with the grid resting upon it and set this in a spot plate depression to allow evaporation of any solvent adhering to the grid. The grid is now ready for analysis or storage.

8.4   Electron Microscopic Examination

8.4.1   Microscope Alignment and Magnification Calibration

Following the manufacturer's recommendations carry out the necessary alignment procedures for optimum specimen examination in the electron microscope. Calibrate the routinely used magnifications using a carbon grating replica.

NOTE: Screen magnification is not necessarily equivalent to plate magnification.

8.4.2   Grid Preparation Acceptability

After inserting the specimen into the microscope, adjust the magnification low enough (300X-1000X) to permit viewing complete grid squares. Inspect at least 10 grid squares for fiber loading and distribution, debris contamination, and carbon film continuity.

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Reject the grid for counting if:

1) The grid is too heavily loaded with
fibers to perform accurate counting and
diffraction operations. A new sample
preparation either from a smaller volume of
water or from a dilution with filtered
distilled water must then be prepared.

2) The fiber distribution is noticeably
uneven. A new sample preparation is
required.

3) The debris contamination is too severe
to perform accurate counting and diffrac-
tion operations. If the debris is largely
organic the filter must be ashed and redis-
persed (see 8.5). If it is inorganic, the
sample must be diluted and again prepared.

4) The majority of grid squares examined
have broken carbon films. A different grid
preparation from the same initial filtra-
tion must be substituted.

8.4.3   Procedure for Fiber Counting

Two methods are commonly used for fiber
counting. In one method (A), 100 fibers
contained in randomly selected fields of
view are counted. The number of fields
plus the area of a field of view must be
known when using this method. In the other
method (B), all fibers (at least 100) in
several grid squares or 20 grid squares are
counted. The number of grid squares
counted and the average area of one grid
square must be known when using this method.

NOTE: The method to use depends upon the
fiber loading on the grid and it is left to
the judgment of the analyst to select the
optimum method. The following guidelines
can be used: If it is estimated that a
grid square (80 μm x 80 μm) contains 50 to
100 fibers at a screen magnification of
20000X, it is convenient to use the field-
of-view counting method. If the estimate
is less than 50, the grid square method of
counting should be chosen. On the other

hand, if the fiber count is estimated to be over 300 fibers per grid square, a new grid containing fewer fibers must be prepared (through dilution or filtration of a smaller volume of water).

8.4.3A Field-of-View Method

After determining that a fiber count can be obtained using this method, adjust the screen magnification to 15,000 to 20,000X. Select a number of grid squares that would be as representative as possible of the entire analyzable grid surface. From each of these squares, select a sufficient number of fields of view for fiber counting. The number of fields of view per grid square is dependent upon the fiber loading. If more than one field of view per grid square is selected, scan the grid opening orthogonally in an arbitrary pattern that prevents overlapping of fields of view. Carry out the analysis by counting, measuring and identifying (see 8.4.4) approximately 50 fibers on each of two grids.

The following rules should be followed when using the field of view method of fiber counting. Although these rules were derived for a circular field of view they can be modified to apply to square or rectangular designs.

1)   Count all fibers contained within the counting area and not touching the circum-ference of the circle.

2)   Designate the upper right-hand quadrant as I and number in clockwise order. Count all fibers touching or intersecting the arc of quadrants I or IV. Do not count fibers touching or intersecting the arc of quadrants II or III.

3)   If a fiber intersects the arc of both quadrants III and IV or I and II count it only if the greater length was outside the arc of quadrants IV and I, respectively.

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4) Count fibers intersecting the arc of both quadrants I and III but not those intersecting the arc of both II and IV.

These rules are illustrated in Fig. 2.

8.4.3B   Grid Square Method

After determining that a fiber count can be obtained using this method adjust the screen magnification to 15,000 to 20,000X. Position the grid square so that scanning can be started at the left upper corner of the grid square. While carefully examining the grid, scan left to right, parallel to the upper grid bar. When the perimeter of the grid square is reached, adjust the field of view down one field width and scan in the opposite direction. The tilting section of the fluorescent screen may be used conveniently as the field of view. Examine the square until all the area has been covered. The analysis should be carried out by counting, measuring, and identifying (see 8.4.4) approximately 50 fibers on each of two grids or until 10 grid squares on each of two grids have been counted. Do not count fibers intersecting a grid bar.

8.4.4   Measurement and Identification

Measure and record the length and width of each fiber having an aspect ratio greater than or equal to three. Disregard obvious biological-bacteriological fibers and diatom fragments. Examine the morphology of each fiber using optical viewing if necessary. Tentatively identify, by reference to the UICC standards, chrysotile or possible amphibole asbestos. Attempt to obtain a diffraction pattern of each fiber utilizing the shortest camera length possible. Move the suspected fiber image to the center of the screen and insert a suitable selected area aperture into the electron beam so that the fiber image, or a portion of it, is in the illuminated area. The size of the aperture and the portion of the fiber should be such that particles other than the one to be examined are
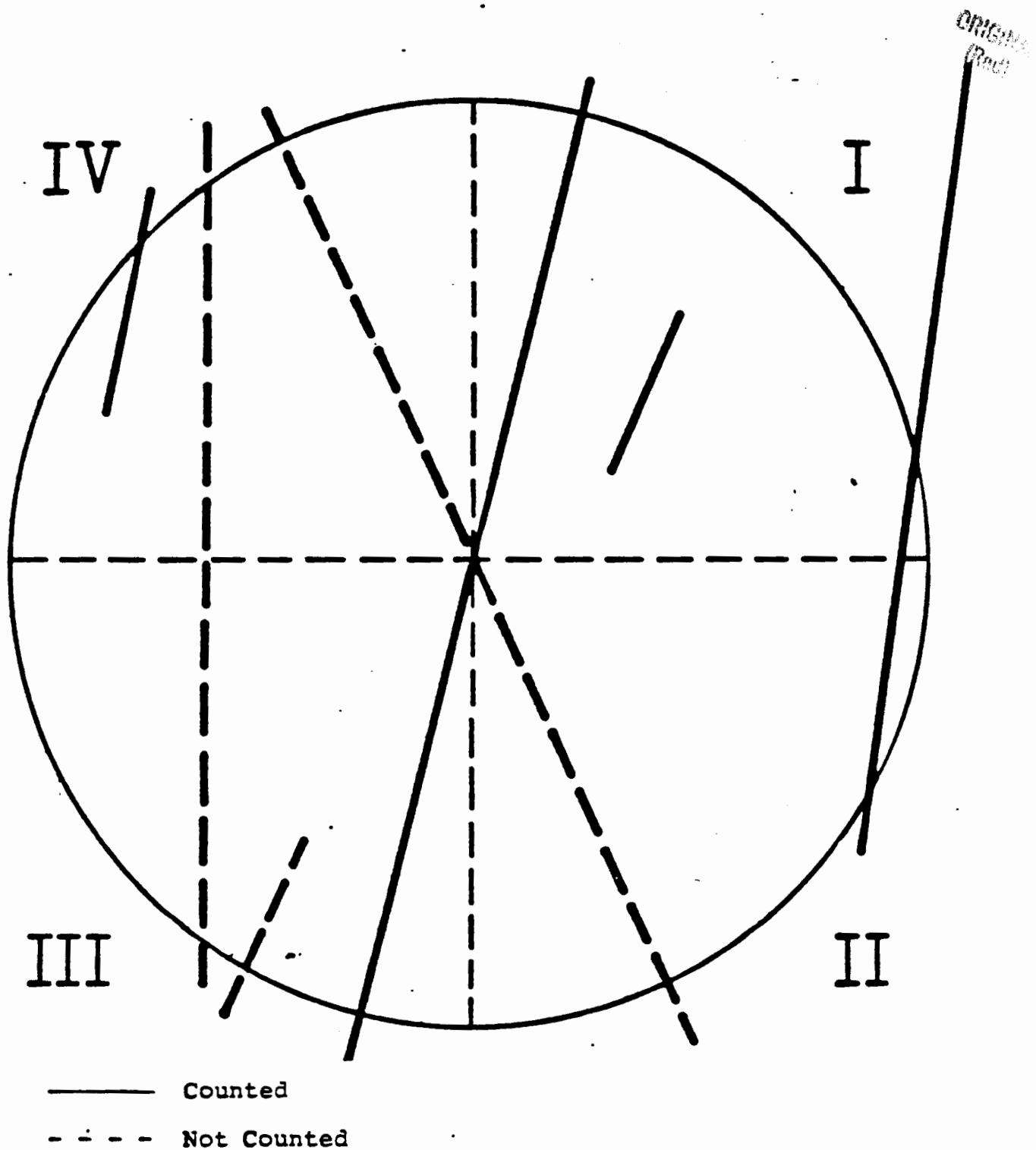
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Figure 2. Illustration of Counting Rules for
Field of View Method.

excluded from the selected area. Observe
the diffraction pattern with the binocular
attachment. If an incomplete diffraction
pattern is obtained, move the particle
image around in the selected area to get a
clearer diffraction pattern or to eliminate
possible interferences from neighboring
particles.

Determine whether the fiber is chrysotile
or an amphibole by comparing the diffrac-
tion pattern obtained to the diffraction
patterns of known standard asbestos
fibers. Confirm the tentative identifica-
tion of chrysotile and amphibole asbestos
from their electron diffraction patterns.
Classify each fiber as chrysotile, amphi-
bole, non-asbestos, no diffraction, or
ambiguous.

NOTE 1: It is convenient to use a tape
recorder during the examination of the
fibers to record all pertinent data. This
information can then be summarized on data
sheets or punched cards for subsequent
automatic data processing.

NOTE 2: Chrysotile fibers occur as single
fibrils or in bundles. The fibrils
generally show a tubular structure with a
hollow canal, although the absence of the
canal does not rule out its identifica-
tion. Amphibole asbestos fibers usually
exhibit a lath-like structure with irreg-
ular ends, but occasionally will resemble
chrysotile in appearance.

NOTE 3: The positive identification of
asbestos by electron diffraction requires
some judgment on the part of the analyst
because some fibers give only partial
patterns. Chrysotile shows unique promi-
nent streaks on the layer lines nearest the
central one and a triple set of double
spots on the second layer line. The
streaks and the set of double spots are the
distinguishing characteristics of chryso-
tile required for identification. Amphi-
bole asbestos requires a more complete
diffraction pattern to be positively iden-
tified. As a qualititative guideline,

layer lines for amphibole, without the
unique streaks (some streaking may be
present) of chrysotile, should be present
and the arrangement of diffraction spots
along the layer lines should be consistent
with the amphibole pattern. The pattern
should be distinct enough to establish
these criteria.

NOTE 4: Chrysotile and thin amphibole
fibers may undergo degradation in an elec-
tron beam; this is particularly noticeable
in small fibers. It may exhibit a pattern
for 1 to 2 seconds and disappear and the
analyst must be alert to note the charac-
teristic features.

NOTE 5: An ambiguous fiber is a fiber that
gives a partial electron diffraction
pattern resembling asbestos, but is insuf-
ficient to provide positive identification.

8.4.5   Determination of Grid Square Area

Measure the dimensions of several represen-
tative grid squares from each batch of
grids with an optical microscope. Calcu-
late the average area of a grid square.
This should be done to compensate for
variability in grid square dimensions.

8.5   Ashing

Some samples contain sufficiently high levels of
organic material that an ashing step is required
before fiber identification and counting can be
carried out.

Place the dried Nuclepore filter paper containing
the collected sediment into a glass vial (28 mm
diameter x 80 mm high). Position the filter such
that the filtration side touches the glass wall.
Place the vial in an upright position in the low
temperature asher. Operate the asher at 50 watts
(13.56 MHz) power and 2 torr oxygen pressure. Ash
the filter until a thin film of white ash remains.
The time required is generally 6 to 8 hours. Allow
the ashing chamber to slowly reach atmospheric
pressure and remove the vial. Add 10 ml of
filtered distilled water to the vial. Place the
vial in an ultrasonic bath for 30 minutes to
disperse the ash. Dilute the sample if required.

Assemble the 25-mm diameter filtering apparatus.
Center a 25-mm diameter 0.1-μm Nuclepore filter
(with the 0.45-μm Millipore backing) on the glass
frit. Apply suction and recenter the filter if
necessary. Attach the filter funnel and turn off
the suction. Add the water containing the
dispersed ash from the vial to the filter funnel.
Apply suction and filter the sample. After drying,
this filter is ready to be used in preparing sample
grids as in 8.3.

NOTE 1: In specifying a 25-mm diameter filter it
is assumed that the ashing step is necessary mainly
because of the presence of organic material and
that the smaller filtering area is desirable from
the point of view of concentrating the fibers. If
the sample contains mostly inorganic debris such
that the smaller filtering area will result in
overloading the filter, the 47-mm diameter filter
should be used.

NOTE 2: It will be noted that a 10-ml volume is
filtered in this case instead of the minimum 50-ml
volume specified in 8.1. These volumes are consis-
tent when it is considered that there is approxi-
mately a 5-fold difference in effective filtration
area between the 25-mm diameter and 47-mm diameter
filters.

NOTE 3: Cross contamination is probable when
ashing more than one sample at a time.

8.6   Determination of Blank Level

Carry out a blank determination with each batch of
samples prepared, but a minimum of one per week.
Filter a fresh supply (500 ml) of distilled,
deionized water through a clean 0.1-μm membrane
filter. Filter 200 ml of this water through a
0.1-μm Nuclepore filter, prepare the electron
microscope grid, and count exactly as in the proce-
dures 8.1 - 8.4. Examine 20 grid squares and
record this number of fibers. A maximum of two
fibers in 20 grid squares is acceptable for the
blank sample.

NOTE: Monitoring the background level of asbestos
is an integral part of the procedure. Upon initia-
ting asbestos analytical work, blank samples must
be run to establish the initial suitability of the
laboratory environment, cleaning procedures, and

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reagents for carrying out asbestos analyses. Analytical determinations of asbestos can be carried out only after an acceptably low level of contamination has been established.

9. Calculations

9.1 Fiber Concentrations

Grid Square Counting Method - If the Grid Square Method of counting is employed, use the following formula to calculate the total asbestos fiber concentration in MFL.

$$C = (\overline{F} \times A_f)/(A_g \times V_o \times 1000) \qquad (1)$$

where: C = Fiber concentration (MFL)

$\overline{F}$ = Average number of fibers per grid opening

A_f = Effective filtration area of filter paper (mm^2) used in grid preparation for fiber counting

A_g = Average area of one grid square (mm^2)

V_o = Original volume of sample filtered (ml)

If ashing is involved, use the same formula but substitute the effective filtration area of the 25-mm diameter filter for A_f instead of that for the 47-mm diameter filter. If one-half the filter is ashed, multiple C by two.

Field-of-View Counting Method - If the Field-of-View Method of counting is employed, use the following formula to calculate the total asbestos fiber concentrations (MFL).

$$C = (\overline{F} \times A_f \times 1000)/(A_v \times V_o) \qquad (2)$$

where: C = Fiber concentration

$\overline{F}$ = Average number of fibers per field of view

A_f = Effective filtration area of filter paper (mm^2) used in grid preparation for fiber counting

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A_V = Area of one field of view (μm^2)

V_O = Original volume of sample filtered (ml)

If ashing is involved, use the same formula but substitute the effective filtration area of the 25-mm diameter filter for A_f instead of that for the 47-mm diameter filter.

9.2   Estimated Mass Concentration

Calculate the mass (μg) of each fiber counted using the following formula.

$$M = L \times W^2 \times D \times 10^{-6}$$

If the fiber content is predominantly chrysotile, the following formula may be used.

$$M = \frac{\pi}{4} \times L \times W^2 \times D \times 10^{-6} \tag{3}$$

where: M = Mass (μg)

L = Length (μm)

W = Width (μm)

D = Density of fibers (g/cm^3)

Then calculate the mass concentration ($\mu g/l$) employing the following formula.

$$M_c = C \times \bar{M}_f \times 10^6$$

where: M_c = mass concentration ($\mu g/l$)

C = fiber concentration (MFL)

$\bar{M}_f$ = mean mass per fiber (μg)

To calculate $\bar{M}_f$ use the following formula.

$$\bar{M}_f = \sum_{i=1}^{n} M_{i/n} \tag{4}$$

where: M_i = mass of each fiber, respectively

n = number of fibers counted

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NOTE 1: Because many of the amphibole fibers are lath shaped rather than square in cross section the computed mass will tend to be high because laths will, in general, tend to lie flat rather than on edge.

NOTE 2: Assume the following densities: chrysotile 2.5, amphibole 3.25.

9.3 Aspect Ratio

The aspect ratio for each fiber is calculated by dividing the length by the width.

10. Reporting

10.1 Report the following concentration as MFL for sample and blank using 95% confidence intervals.

a. Chrysotile

b. Amphibole

c. Total asbestos fibers

10.2 Use two significant figures for concentrations greater than 1 MFL, and one significant figure for concentrations less than 1 MFL.

10.3 Tabulate the size distribution, length and width.

10.4 Tabulate the aspect ratio distribution.

10.5 Report the calculated mass as μg/1.

10.6 Indicate the detection limit in MFL.

10.7 Indicate if less than five fibers were counted.

10.8 Include remarks concerning pertinent observations, (clumping, amount of organic matter, debris) amount of suspected though not identifiable as asbestos fibers (ambiguous).

11. Precision

11.1 Intra-Laboratory

The precision that is obtained within an individual laboratory is dependent upon the number of fibers counted. If 100 fibers are counted and the loading

is at least 3.5 fibers/grid square, computer
modeling of the counting procedure shows that a
relative standard deviation of about 10% can be
expected.

In actual practice some degradation from this
precision will be observed but should not exceed ±
15% if several grids are prepared from the same
filtered sample. The relative standard deviation
of analyses of the same water sample in the same
laboratory will increase as a result of sample
preparation errors and a relative standard devia-
tion of about about ± 25 to 35% will occur. As the
number of fibers counted decreases, the precision
will also decrease approximately proportional to $\sqrt{N}$
where N is the number of fibers counted. The
precision for mass concentration is generally
poorer than that for fiber concentration.

Based upon the analysis of one laboratory utilizing
a different analyst for each of three water
samples, intra-laboratory precision data are
presented in Table 1.

11.2   Inter-Laboratory

Based upon the analysis by various government and
private industry laboratories of filters prepared
from nine water samples, inter-laboratory precision
data of the method are presented in Table 2.

12. Accuracy

12.1 Fiber Concentrations

As no standard reference materials are available,
only approximate estimates of the accuracy of the
procedure can be made. At 1 MFL, it is estimated
that the results should be within a factor of 10 of
the actual asbestos fiber content.

This method requires the positive identification of
a fiber to be asbestos as a means for its quantita-
tive determination. As the state-of-the-art
precludes the positive identification of all of the
asbestos fibers present, the results of this
method, as expressed as MFL, will be biased on the
low side and, assuming no fiber loss, represent 0.4
to 0.8 of the total asbestos fibers present.

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12.2 Mass Concentrations

As in the case of the fiber concentrations, no standard samples of the size distribution found in water are available. The estimated mass concentration is often very inaccurate because of poor counting statistics associated with large fibers that are few in number but represent most of the actual mass concentration.

13. Suggested Statistical Evaluation of Grid Fiber Counts

13.1 Because the fiber distribution on the sample filter, resulting from the method of filtration, has not been fully characterized, the fiber distribution obtained on the electron microscope grids for each sample should be tested statistically • against an assumed distribution and a measure of the precision of the analysis should be provided.

13.2 Assume that the fibers are uniformly and randomly distributed on the sample filter and grids. One method for confirming this assumption is given below.

Using the chi-square test, determine whether the total number of fibers found in individual grid openings are randomly and uniformly distributed among the openings using the following formula.

$$X^2 = \sum_{i=1}^{N} \frac{(n_i - np_i)^2}{np_i} \qquad (5)$$

where: X^2 = Chi-square statistic

N = Number of grid openings examined for the sample

n_i = Total number of fibers found in each respective grid opening

n = Total number of fibers found in N grid openings

p_i = Ratio of the area of each respective grid opening to the sum of the areas of all grid openings examined

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NOTE: If an average area for the grid squares has been measured as outlined in 8.4.5, the term np_i represents the mean fiber count per grid square.

If the value for X^2 exceeds the value listed in statistical tables for the 0.1% significance level with N-1 degrees of freedom, the fibers are not considered to be uniformly and randomly distributed among the grid openings. In this case, it is advisable to try to improve the uniformity of fiber deposition by filtering another aliquot of the sample and repeating the analysis.

13.3   If uniformity and randomness of fiber deposition on the microscope grids has been demonstrated as in 13.2, and the fiber concentration is assumed to be normally distributed about the mean value, the 95% confidence interval about the mean fiber concentrations for chrysotile, amphibole, and total asbestos fibers may be determined using the following formulae.

$$S_c = \left[\frac{N \sum_{i=1}^{N} X_i^2 - (\sum_{i=1}^{N} X_i)^2}{N(N-1)} \right]^{1/2} \tag{6}$$

where: S_c = Standard deviation of the chrysotile fiber count

   N = Number of grid openings examined for the sample

   X_i = Number of chrysotile fibers in each grid opening, respectively

Obtain the standard deviations of the fiber counts for amphibole asbestos fibers and for total asbestos fibers by substituting the corresponding value of X_i into equation (6).

$$X_u = \bar{X} + \frac{tS_c}{\sqrt{N}} \tag{7}$$

$$X_L = \bar{X} - \frac{tS_c}{\sqrt{N}} \tag{8}$$

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12.2 Mass Concentrations

As in the case of the fiber concentrations, no standard samples of the size distribution found in water are available. The estimated mass concentration is often very inaccurate because of poor counting statistics associated with large fibers that are few in number but represent most of the actual mass concentration.

13. Suggested Statistical Evaluation of Grid Fiber Counts

13.1 Because the fiber distribution on the sample filter, resulting from the method of filtration, has not been fully characterized, the fiber distribution obtained on the electron microscope grids for each sample should be tested statistically against an assumed distribution and a measure of the precision of the analysis should be provided.

13.2 Assume that the fibers are uniformly and randomly distributed on the sample filter and grids. One method for confirming this assumption is given below.

Using the chi-square test, determine whether the total number of fibers found in individual grid openings are randomly and uniformly distributed among the openings using the following formula.

$$X^2 = \sum_{i=1}^{N} \frac{(n_i - np_i)^2}{np_i} \tag{5}$$

where: X^2 = Chi-square statistic

N = Number of grid openings examined for the sample

n_i = Total number of fibers found in each respective grid opening

n = Total number of fibers found in N grid openings

p_i = Ratio of the area of each respective grid opening to the sum of the areas of all grid openings examined

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TABLE 1. INTRA-LABORATORY PRECISION

Sample Type	Number of Sample Aliquots Analyzed	Mean Fiber Concentration MFL (millions of asbestos fibers/l)	Precision, Relative Standard Deviation	Mass Concentration (μg/l)	Precision, Relative Standard Deviation
Chrysotile (UICC)	26	23	37%	0.32	71%
Crocidolite (UICC)	20	8	36%	1.5	48%
Taconite (raw water)	20	16	24%	10.5	65%

TABLE 2. INTER-LABORATORY PRECISION

Sample Type	Number of Labs Reporting	Mean Fiber Concentration, MFL (millions of asbestos fibers/l)	Precision, Relative Standard Deviation
Chrysotile	10	877	35%
	9	119	43%
	11	59	41%
	9	31	65%
	9	28	32%
	3	25	35%
Amphibole	11	139	50%
	4	95	52%
	14	36	66%

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where: X_u = Upper value of 95% confidence interval for chrysotile

X_L = Lower value of 95% confidence interval for chrysotile

$\bar{X}$ = Average number of fibers per grid opening

t = Value listed in t-distribution tables at the 95% confidence level for a two tailed distribution with N-1 degree of freedom

S_c = Standard deviation of the fiber counts for chrysotile

N = Number of grid openings examined for the sample

The values of X_u and X_L can be converted to concentrations in millions of fibers per liter using the formula in section 9 and substituting either X_u or X_L for the term F.

Obtain the upper and lower values of the 95% confidence interval for amphibole asbestos fibers and total asbestos fibers by substituting the corresponding values of $\bar{X}$ and S into equations (7) and (8).

Report the precision of the analysis, in terms of the upper and lower limits of the 95% confidence interval, for chrysotile, amphibole, and total asbestos fiber content. If a lower limit is found to be negative, report the value of the limit as zero.

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15. SUPPLEMENTARY NOTES

16. ABSTRACT

This manual describes an interim electron microscope (EM) procedure for measuring the concentration of asbestos in water samples. The main features of the method include filtering the sample through a sub-micron polycarbonate membrane filter, examining an EM specimen grid in a transmission electron microscope (TEM), and verifying fiber identity by selected area electron diffraction (SAED).

This interim method is a revision of the procedure issued in 1976 and reflects the improvements that have been made in asbestos analytical methodology since that time.

17. KEY WORDS AND DOCUMENT ANALYSIS

a. DESCRIPTORS	b. IDENTIFIERS/OPEN ENDED TERMS	c. COSATI Field/Group
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October 4, 1988

Mr. Paul Racette (3HW23)
Site Investigation and Support Section
U.S. EPA Region III
841 Chestnut Building
Philadelphia, PA 19107

Subject: Site Inspection - Ametek, Inc.

Dear Mr. Racette:

The Department of Natural Resources and Environmental Control (DNREC) is
planning on conducting a Site Inspection (SI) at Ametek, Inc. on October
18, 1988. DNREC proposes to sample the soils, surface water and sediments.
The sample locations will be determined at the time of the SI.

Attached is a map showing the expected ground water flow direction. Please
contact Brad L. Smith of my staff with any questions or comments.

Sincerely,

Joseph J. Hardman, P.E.
Supervisor

JJH:JH:dls
JH2023

Attachment

cc: P. G. Retallick
     G. A. Molchan
     B. L. Smith
     J. Hackney

ORIGINAL
(RED)

AVONDALE, PA. 8 MI.
HOCKESSIN 4 MI.

40' | ⁴43
⁴45
440 000 FEET |
75°37'30"
39°45'

ELSMERE 1.9 MI.
WILMINGTON (CITY HALL) 4.8 MI.
NEWPORT 1.5 MI.
NEW CASTLE 6.1 MI.

Bissell Sanitorium
County Farm
Golf Course
X 245
CASTLE RECREATION AREA
Faulkland
Brandywine Springs Park
Run
Hyde
Cedars
Clay
Creek
Clanton
Milltown
2
B & O
Substation
630 000 FEET
Cem
Substa
Marshallton
Kiamensi
Ford (Powell) Park
Red
4397

Ametek, Inc.

Expected
Groundwater
Flow Direction

TRAIL
OHIO
Call Run South Park
St James Ch.
Cem
Trailer Park
Stanton
Trailer Park
Drive-in Theater
BALTIMORE
BM
55
4
4396
Pleasant Hills Park
Island
Cheese
and
Bread
Glenville Park
Water
Sewage Disposal
Oil
42'30"
Delaware Park Race Track
AMTRAK
4 TRACKS
White
Clay
Creek
4395
N 5 MI.